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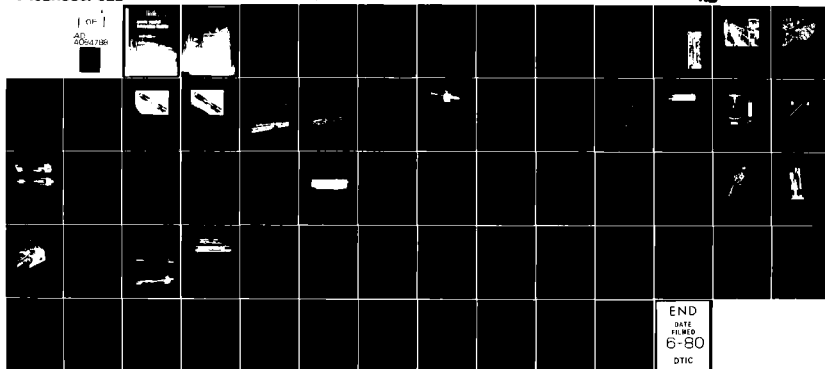
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The report describes the design and operation of a five chamber, interconnected vacuum system, which is capable of cleaning, plating, and sealing precision quartz crystal units in ceramic flatpack enclosures continuously in a high vacuum environment. The production rate design goal was 200 units per eight hour day. → next page		

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20. ABSTRACT (continued)

A unique nozzle beam gold deposition source was developed to operate for extended periods of time without reloading. The source puts out a narrow beam of gold typically in the order of  $2\frac{1}{2}^\circ$  included "cone" angle. Maximum deposition rates are in the order of 400  $\text{\AA}/\text{min}$  at 5.5 in. "throw" distance used.

Entrance and exit air lock chambers expedite the material throughput, so that the processing chambers are at high vacuum for extended periods of time. A stainless steel conveyor belt, in conjunction with three vacuum manipulators, transport the resonator components to the various work stations. Individual chambers are normally separated from each other by gate valves. The crystal resonators, mounted in flatpack frames but unplated, are loaded into transport trays in a lid-frame-lid sequence for insertion into the system and exit as completed crystal units.

The system utilizes molybdenum disulfide coated ball bearings at essentially all friction surfaces. The conveyors are capable of operating at  $300^\circ\text{C}$  temperature and  $10^{-8}$  Torr. Metal bellows or magnetic drives are utilized to transfer motion into the vacuum chambers.

The gold sources and plating mask heads are equipped with elevators and gate valves, so that they can be removed from the system for maintenance without exposing the chambers to atmosphere.

The work stations include: ultraviolet/ozone cleaning,  $300^\circ\text{C}$  vacuum baking, coarse and fine gold deposition stations with automatic frequency plating controls, and a gold thermocompression sealing station. The length of the combined vacuum envelope is 24 feet.

## PREFACE

This is the second report on a manufacturing methods and technology (MM&T) program funded by the U. S. Army Electronics Research and Development Command (ERADCOM), Fort Monmouth, New Jersey, covering a period from April 1, 1977 to September 30, 1979.

The work is being performed by the Neutron Devices Department of General Electric Company, (GEND) located at St. Petersburg, Florida, a contractor of the U. S. Department of Energy. This facility is government owned.

ERADCOM requested that the described work be done by this Department of Energy facility because no bids had been received by ERADCOM in response to its multiple source solicitation (including advertisement in the Commerce Business Daily) to have this work performed at an industrial facility. The government would prefer to have all future projects, especially the eventual production of the ceramic flatpack crystal units, performed at a qualified commercial facility. In connection with this, an industry demonstration of the pilot line industry facility is being planned, tentatively for sometime during 1981. The date of this demonstration will be advertised in Commerce Business Daily. Those who wish to be notified directly of the date should contact:

U. S. Army Electronics Technology and Devices Laboratory  
ATTN: DELET-MQ (Dr. John R. Vig)  
Fort Monmouth, NJ 07703

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## INTRODUCTION

This report describes the results of Phase I of a manufacturing methods and technology (MM&T) program aimed at establishing a pilot line for producing high precision crystals. The work is continuing under Phases II and III. Phase I was aimed at designing and building an in-line ultrahigh vacuum quartz crystal fabrication facility (QXFF) for the final steps (i.e., cleaning, baking, plating and sealing) in the fabrication of precision crystals.<sup>1</sup> Phase II is aimed at establishing a pilot line for 22-MHz high shock crystals in a small (HC-18 equivalent) ceramic flatpack. Phase III is aimed at establishing a pilot line for 5- to 10-MHz crystals in a larger (HC-6 equivalent) ceramic flatpack.

As indicated in the first report,<sup>2</sup> the nozzle beam gold source was perceived as probably the most difficult engineering task of the Quartz Crystal Fabrication Facility project. For this reason, effort was concentrated on developing and testing the nozzle beam source.

In addition, prototypes of the remaining critical components of the system were designed, built, and tested. The heretofore untested critical components were: the mask, the mask head, the elevators, and the sealing mechanism. After testing and making modifications, the appropriate quantity of each item was procured.

The vacuum system envelope and the support structure were also designed and procured. The vacuum system was assembled and operated.

The electric power supplies, temperature controllers, cryopump compressors, and mass spectrometer indicator/controller were placed in cabinets. The frequency plating control equipment was designed and built.

## VACUUM SYSTEM ENVELOPE

The vacuum system, as described in the first report, was built by Perkin-Elmer Ultek, Inc. The entire vacuum envelope complied with the initial specifications, as received, except for one small vacuum leak. The vacuum envelope, as well as all other components of the system, are shown in the schematic cross-sectional diagram of the facility (Figure 1). Photographs of the complete envelope and facility are shown in Figures 2, 3 and 4. The total length of the assembled chambers is about 24 feet.

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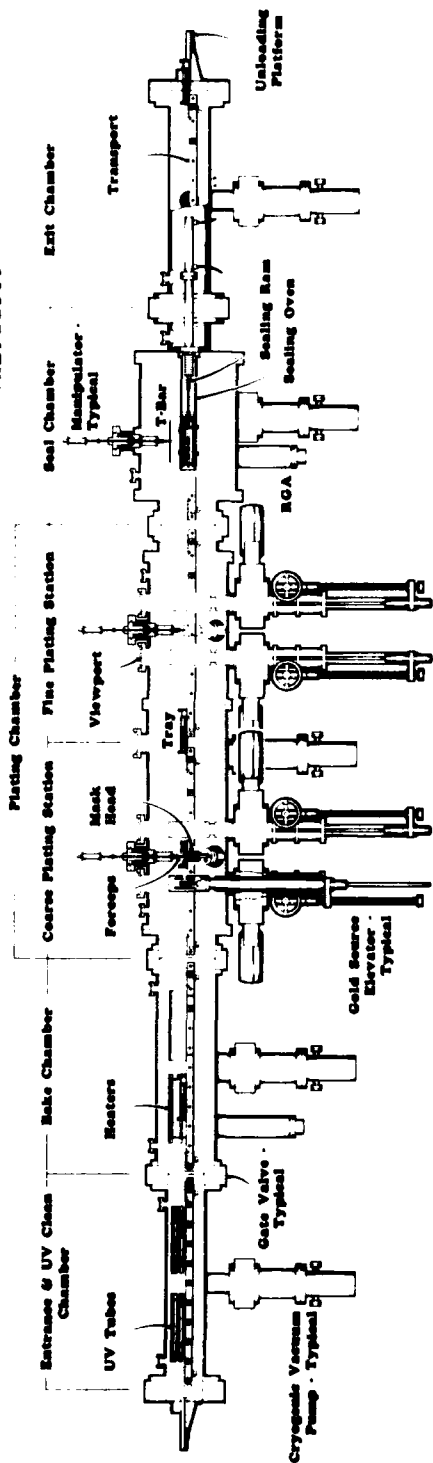


Figure 1. Quartz Crystal Fabrication Facility, Schematic

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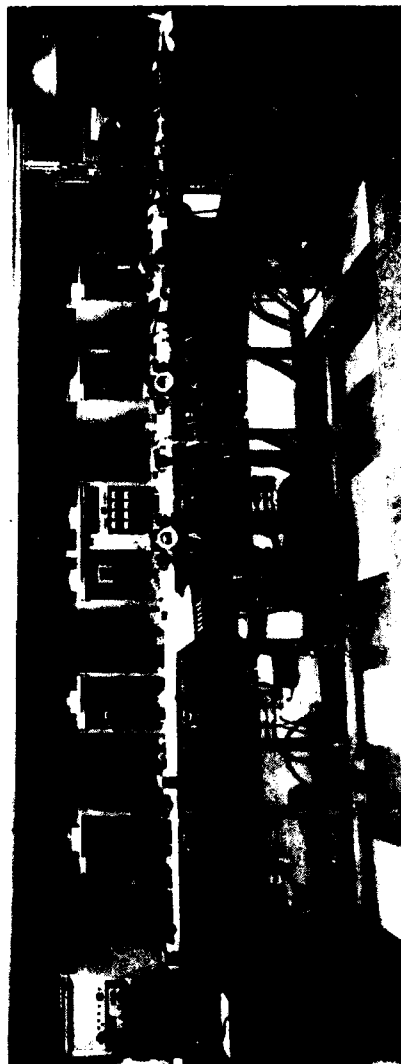


Figure 2. Quartz Crystal Fabrication Facility, Front View

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Figure 3. Quartz Crystal Fabrication Facility, Front Oblique End View



Figure 4. Quartz Crystal Fabrication Facility, Back Oblique End View

The system contains five individual "in-line" chambers, separated by gate valves. Each chamber is individually pumped by cryogenic pumps; rough pumping is accomplished by cryo-sorption pumps.

#### LOADING/ULTRAVIOLET CLEAN CHAMBER

The first chamber is an airlock chamber; it enables the loading of material into the bake chamber without exposing that chamber to atmosphere. It also serves as an ultraviolet/ozone cleaning chamber. This chamber contains two banks of low pressure mercury/UV tubes, mounted over the transport. The first bank of tubes has an output at 1849 Å and 2537 Å, and is used to clean the inserted materials by the UV/ozone cleaning method.<sup>3</sup>

The second bank of tubes has an output of 2537 Å only, and is used to decompose ozone. Safety practices require that ozone not be pumped, nor flushed into the atmosphere. The UV tubes have a special dual envelope that provides extra protection against accidental mercury contamination of the system. The chamber was designed for the throughput of one tray per hour.

#### BAKE CHAMBER

The second chamber is the bake chamber; it contains two banks of tantalum heaters mounted over the transport. Each bank is capable of heating one tray of crystals up to 300°C. The chamber pressure is typically  $5 \times 10^{-8}$  after a one-hour tray baking cycle. The chamber was designed for a throughput of one tray per hour.

#### PLATING CHAMBER

The third chamber is the plating chamber. It contains two gold plating stations; one for coarse plating and another for fine plating. The coarse plating can be started at about 200°C crystal temperature, while the fine plating is done typically at the upper turnover temperature for AT-cut crystals. The coarse plating station contains a tray temperature holding oven. This oven is used to minimize "heat shock" on the crystals going into the heated plating head. It also increases the speed of operation.

There is a need to cool the crystals between the coarse and fine plating stations. Place is provided for two trays on each transport between the two stations.

Experiments indicate that crystal frames at about 300°C, placed into the fine plating head, will cool to within 5°C of the upper turning temperature in 2 1/2 minutes. Cooling in the tray for one hour, from the initial 300°C temperature, results in a 2 min cooling time in the fine plating head. However, the thermal shock to the crystal is greatly reduced in the second case.

The throughput of the coarse plating station is described under the heading "Evaporant Source."

#### SEALING CHAMBER

The fourth chamber is the sealing chamber; it contains a preheat furnace, a ram and furnace for thermo-compression sealing,<sup>1</sup> and a tray transfer manipulator.

The preheat furnace is used to minimize the thermal shock to the crystal at insertion into the sealing furnace and also to help maintain the throughput of the system.

The sealing chamber has provisions for gas back filling of the crystal units, when specified. This chamber has a throughput of one tray per hour. The pressure during the sealing operation at 300°C oven temperature was typically  $5 \times 10^{-7}$  Torr. Residual gas data is given in the Appendix (Figure I).

#### EXIT CHAMBER

The exit chamber is an airlock chamber, similar in construction to the entrance chamber. It is used to unload completed crystal units from the system. It has provisions for rough and fine pumping, and it contains one transport.

#### ENVELOPE SUPPORT STRUCTURE

The support structure design is based on the principle of unrestrained platforms in the horizontal plane. The five individual chambers are attached to individual "floating" platforms. Each platform is supported by four ball coasters engaging a ground plate. The elevation of the chamber end flanges are adjusted by shimming of the stand. When the chamber end flanges are bolted together, the external forces are thus substantially eliminated by the "floating" platforms. Thermal expansion during chamber bakeout results in a platform translation, so that substantially no stresses result along the chamber axis. Individual chambers may be separated at the connecting flanges and moved forward for maintenance.

#### RESONATOR TRAY

The tray carrier was built as indicated in the first report (see Figures 5 and 6). Experiments with the tray indicated that, after a few heating cycles to 300°C, the corrugated steel ribbon, serving as the flatpack frame-to-lid spacer, failed. Molybdenum ribbon was substituted for the spring steel stock. After a few dozen operations, even the molybdenum springs failed.

Analysis of the problem indicated that the corrugated springs were overstressed at the 300°C operation. Due to the lack of space in the tray, it was not practical to reduce the stresses in the spring design.

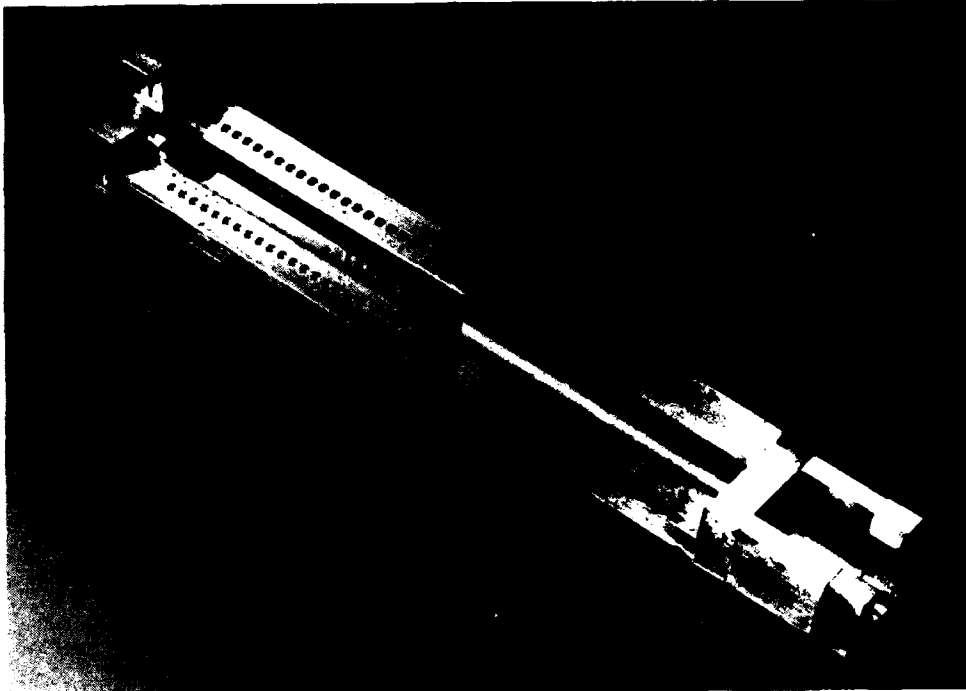


Figure 5. Resonator Tray, Empty

A design utilizing pivoted "fingers" was then arrived at. In this design, each row of fingers is loaded with two tungsten flat springs. These springs are stressed considerably below the yield strength of tungsten at 300°, so that no spring failure should occur. Indeed, experiments indicated that these trays survived at least 36 production runs without any sign of deterioration.

These pivoted fingers are well restricted in their motion and, unlike the corrugated springs, will not get "caught" between the last lid and the stationary anvil.

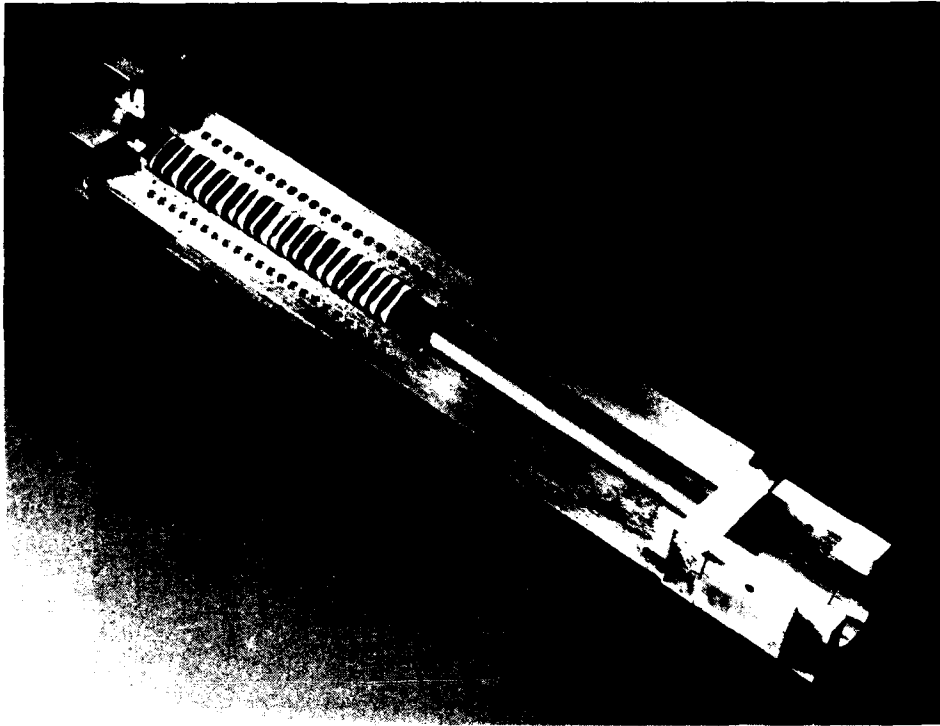


Figure 6. Resonator Tray, Loaded

The initial trays contained separators for 10 crystal units, but were long enough for the addition of spacers to hold 25 units. The first attempt at sealing with 20 units simultaneously was accomplished with 100 percent yield at Radiflo\* leak detection.

The entire system is designed so that the ultraviolet tubes, heaters, and the sealing oven/ram, are capable of accommodating trays with 25 crystal units.

\*Trademark, Iso Vac Engineering



## TRAY TRANSPORT

The tray transport was built substantially as described in the first report (see Figure 7). Two types of tests were conducted on the transport after installation into the chambers:

1. Temperature (300°C) and continuous vacuum operation,
2. Transport-to-transport tray transfer operation (see Figure 8).

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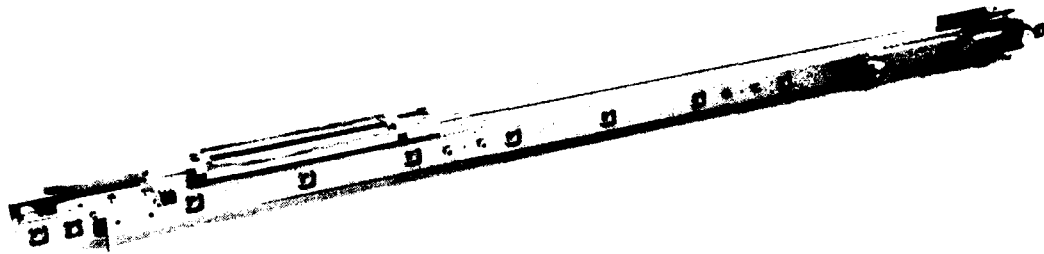


Figure 7. Tray Transport

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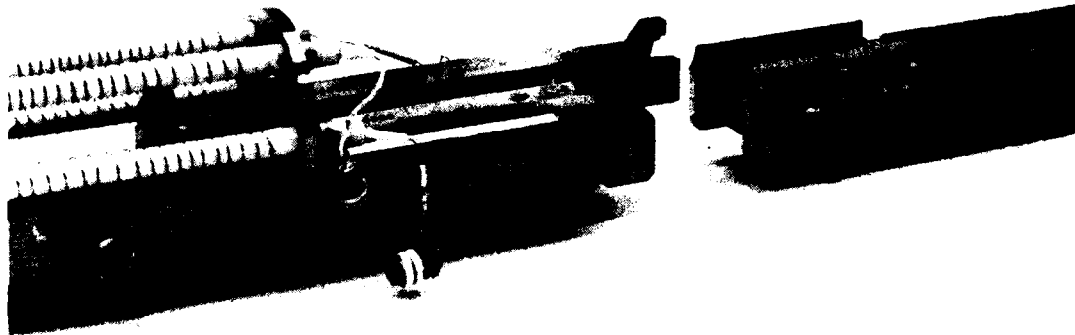


Figure 8. Tray Transfer Operation

Extended time period operations of the transport were made in the bake chamber under continuous vacuum of  $10^{-7}$  to  $10^{-8}$  Torr, with the heaters operating at  $300^{\circ}\text{C}$  crystal temperature for about eight hours per day. After three to four days of this operation, the commercial magnetic feedthrough drive shaft started to run "rough". Within about ten days of operation under the above conditions, the shaft seized up completely. These shafts were mounted in molybdenum disulfide coated journal bearings.

The journal bearings in the feedthrough were then replaced by molybdenum disulfide coated ball bearings. No further problems were observed, after over six months of operation.

Transport-to-transport transfer experiments were conducted in the actual chambers. The transfer was effected flawlessly. It was noted, however, that in a few instances the crystals' tray was partially knocked off the transport by a manipulator operator error.

In order to eliminate this problem, dual funnel-shaped guides were placed at the ends of the transports (see Figure 9). These guides are capable of "righting" the misaligned trays on the transport, just by driving the trays through the guides.

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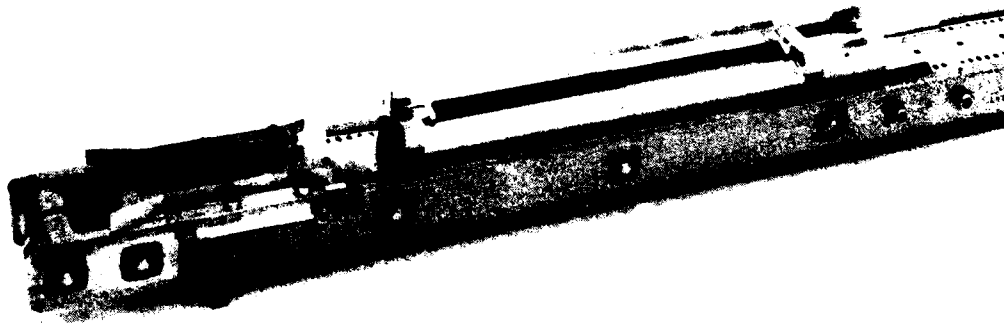


Figure 9. Tray Transport Showing Funnel-Shaped Guides

## VACUUM MANIPULATOR

The three manipulators were built similar to the first model (see Figures 10 and 11), as described in the first report.<sup>1</sup> Operation of the manipulator/forceps in the actual transfer of the crystal frame from the tray to the mask and back to the tray indicated only one flaw. The forceps' jaws did not grip the frame firmly, due to the jaws' pivoted mounting. Also, there was a tendency for the frame to slide back into the forceps, and it was cumbersome to retract the frame from that position.

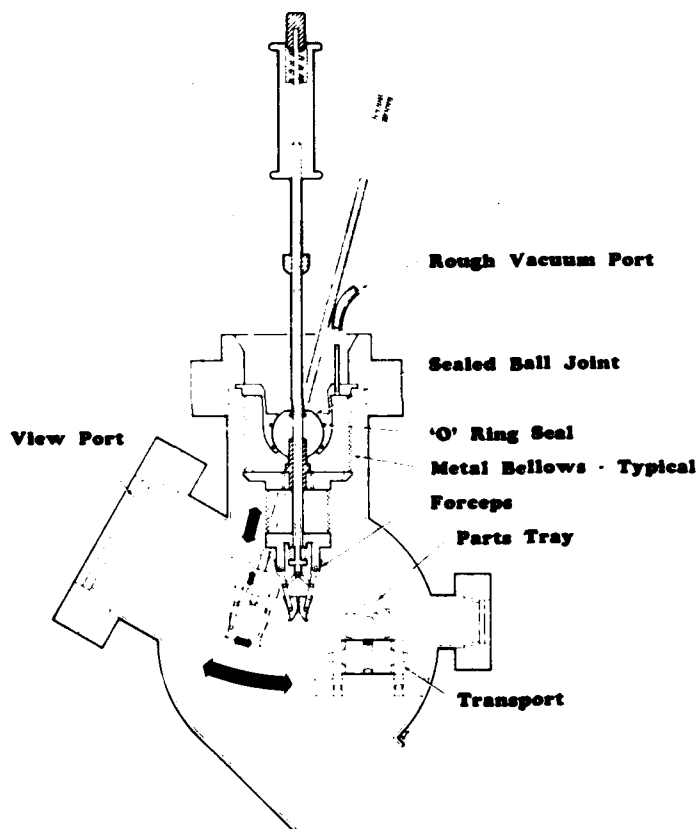


Figure 10. Vacuum Manipulator, Schematic

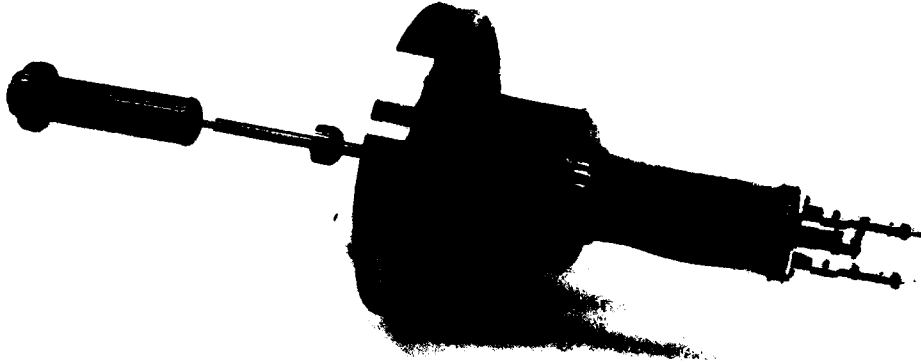
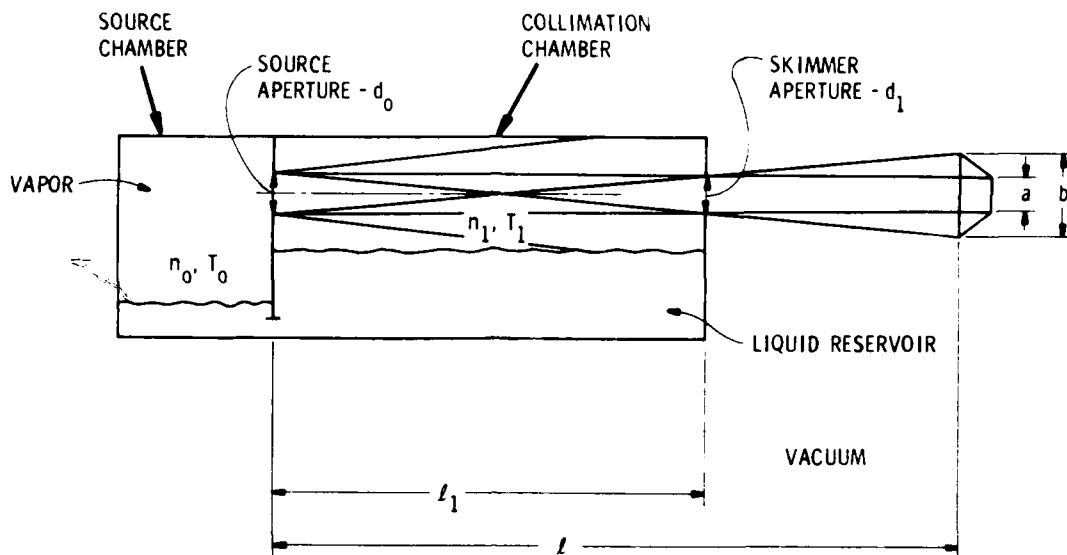


Figure 11. Vacuum Manipulator

This problem was solved by using nonpivoted (i.e., firm) jaws and frame backstops on the jaws. Unfortunately, however, this made the forceps noncompatible with the various size flatpack resonator frames. The use of parallel motion forceps would solve this incompatibility problem.

#### EVAPORANT SOURCE (NOZZLE BEAM)

The first nozzle beam source was fabricated at GEND early in 1977 according to the design by R. P. Andres, E. Hafner and J. R. Vig.<sup>4</sup> A schematic diagram of the classical nozzle beam source is shown in Figure 12. Tungsten was used for both the crucible and source tube. It was found that after the second heating cycle, the crucible cracked. Investigation indicated that since the gold adheres to the walls of the tungsten crucible, and since the gold-to-tungsten thermal expansion ratio is about 3.5:1, cooling permanently deformed both the gold charge and the tungsten crucible walls. At subsequent heating, further permanent deformation was imparted to the tungsten crucible walls due to the higher thermal expansion of the gold. Eventually, the cylindrical walls would fail in the hoop stress cracking mode.



THE CLASSICAL EXPRESSIONS FOR DENSE FLOW ARE:

$$f_0 = (0.513) n_0 \left[ \frac{2kT_0}{m} \right]^{1/2} \left[ \frac{\pi d_0^2}{4} \right] \quad (1)$$

$$\text{AND} \quad I(\theta = 0, l) = f_0 \left[ \frac{1}{\pi l^2} \right] \quad (2)$$

WHERE  $f_0$  = TOTAL SOURCE FLOW (MOLECULES/SEC)

$n_0$  = SOURCE DENSITY (MOLECULES/CC)

$T_0$  = SOURCE TEMPERATURE ( $^{\circ}$ K)

$k$  = BOLTZMANN'S CONSTANT (ERG/ $^{\circ}$ K)

$m$  = MOLECULAR WEIGHT OF VAPOR (GM/MOLECULE)

$d_0$  = SOURCE APERTURE DIAMETER (CM)

$I(\theta = 0, l)$  = CENTERLINE FLUX INTENSITY (MOLECULES/CM<sup>2</sup> SEC)

$l$  = DISTANCE FROM SOURCE APERTURE (CM)

**Figure 12. Nozzle Beam Evaporant Source, Schematic**  
(A plot of Eq. 2 for gold is given in the Appendix, Figure II.)

Other crucible materials were investigated. Graphite appeared to be the best candidate for the following reasons:

1. Low vapor pressure up to 2500°C,
2. Liquid gold does not "wet" graphite, hence it will not run over the side walls of the crucible, and it will not run into gaps between bores and close fitting plugs,
3. Graphite crucibles can be temperature cycled with liquid or solid gold without cracking them,
4. It has relatively high thermal resistance, so that heat zones can be well confined,
5. High purity material is commercially available,
6. It is relatively easy to machine,
7. It has moderately high electrical resistivity, so that practical heating elements may be constructed of graphite.

A crucible and source tube were built using Poco Graphite Inc. "filled pore" and purified DFP 3-2 material. An isometric drawing of a current device is given in Figure 13. The crucible is shown in Figure 14 and the crucible with triple radiation shield in Figure 15. Current sources have five element radiation shields made of tungsten foil.

External heaters were used to melt the gold charge initially, and to maintain crucible temperature. Source tube temperatures were measured by an optical pyrometer through a "window" in the crucible wall. Later, the power input to the source tube was increased by increasing the source tube diameter, so that external heaters are no longer required.

In the early experiments a glass slide was used for the target, permitting observation of the film build-up from the back side. The source tube was operated up to 2400°C at midspan. No visible amount of gold was deposited on the target glass in several minutes at 2400°C. It was soon realized that the liquid gold was pushed out of the source tube and back into the reservoir by the gold vapor pressure at elevated temperatures ( $P \simeq 1$  cm Hg at 2000°C). A tungsten rod was placed into the source tube bore in order to "wick" the gold up into the heat zone of the source tube, and to provide relatively large gold surface area for vaporization. A picture of a current model wick is given in Figure 16. An X-ray photograph of the gold source is shown in Figure 17. This source has clocked over 1,000 hours of beaming. The wick has two axial and thirty

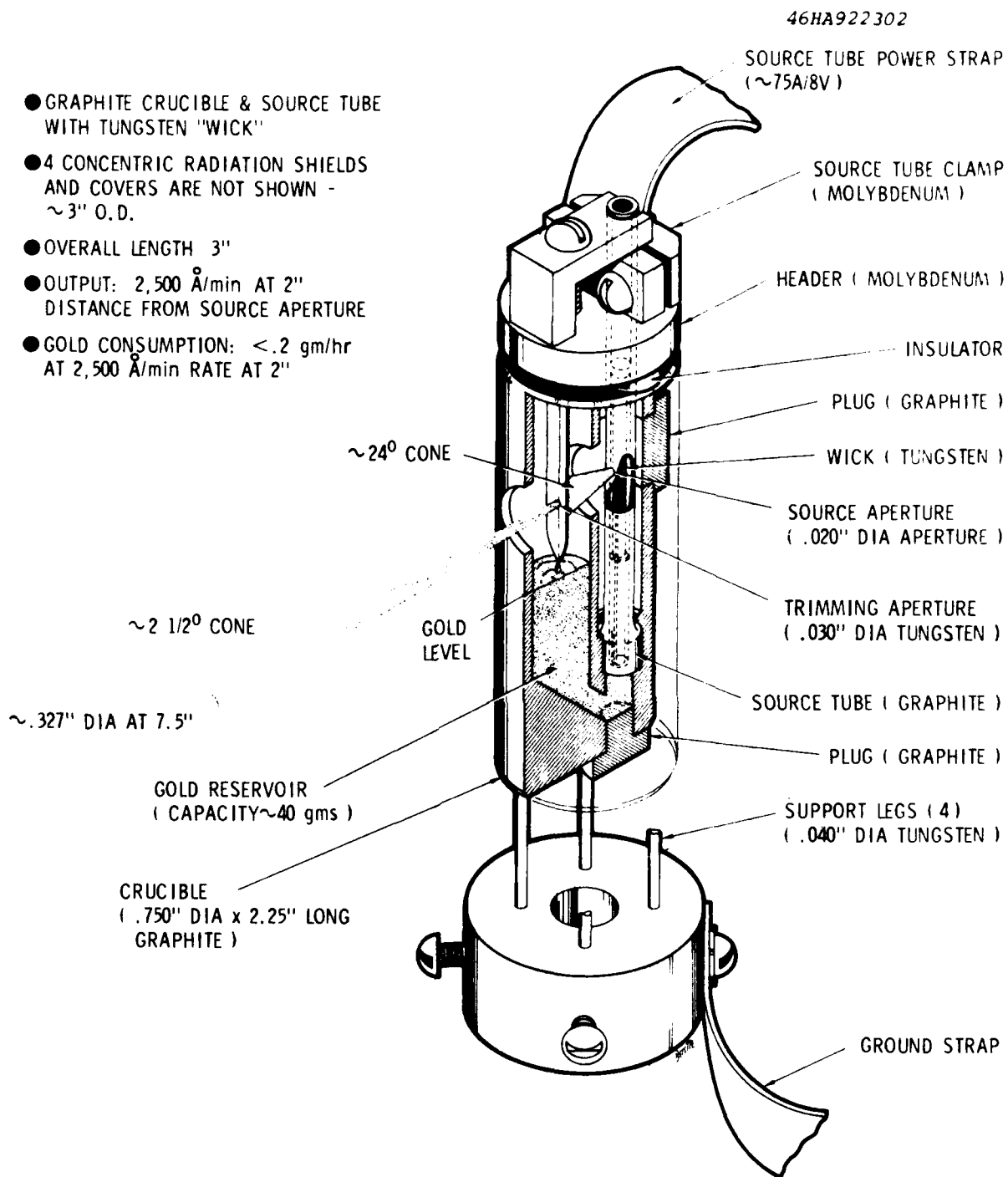


Figure 13. Nozzle Beam Evaporant Source, Isometric

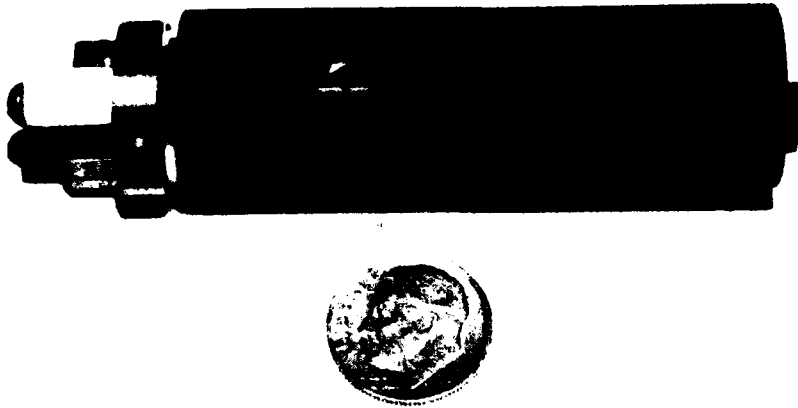


Figure 14. Crucible, Nozzle Beam Evaporant Source

radial grooves on its surface in order to reliably feed the gold over the wick surface. These grooves also increase the surface area over that of a plain rod for increased vaporization rate.

With the wick in the source tube, gold was finally deposited on the target. Initially, the deposition rate was about 40 Å/min at 7.5-in. source tube-to-target spacing.



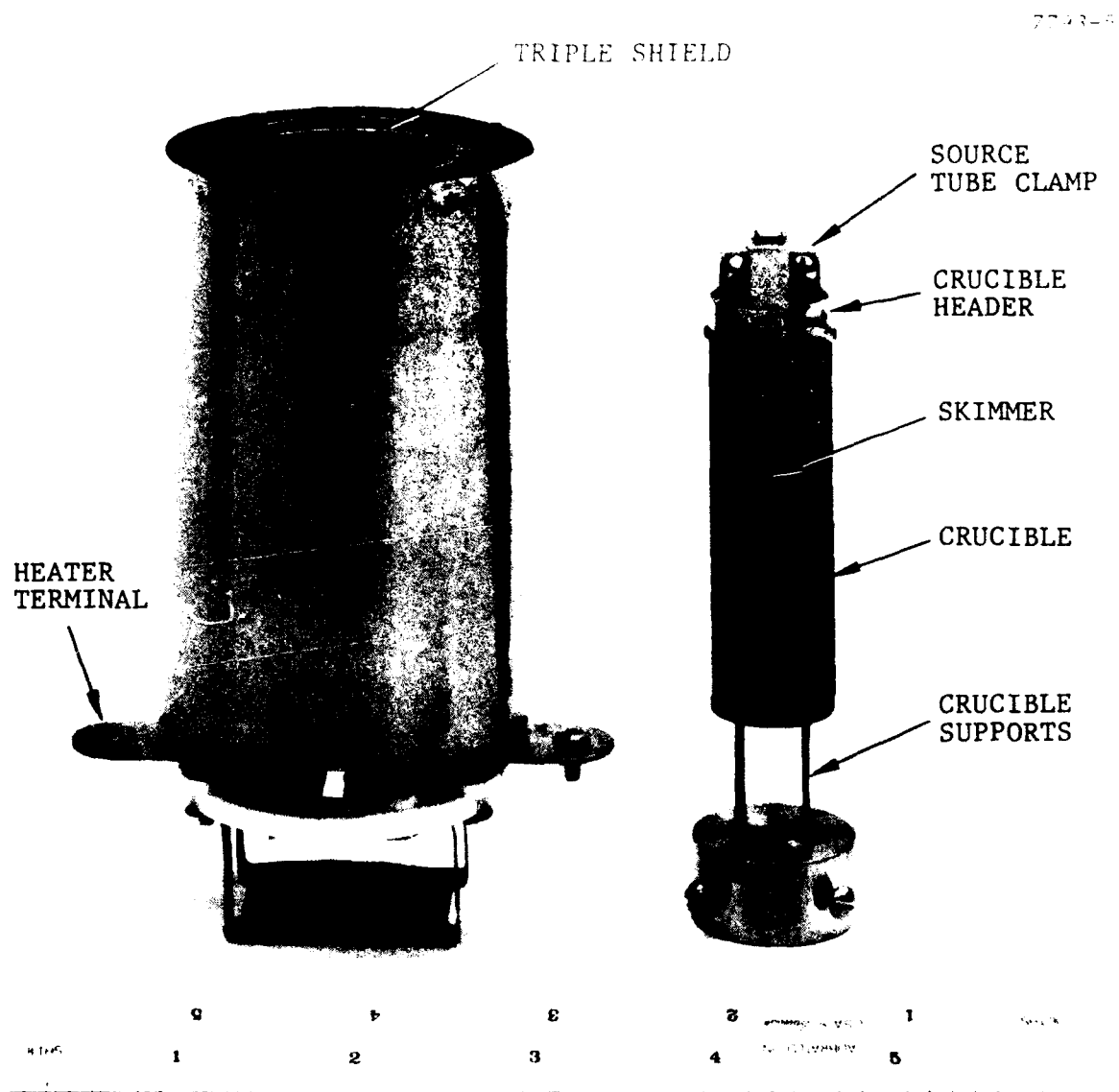


Figure 15. Nozzle Beam Evaporant Source, Crucible and Shield

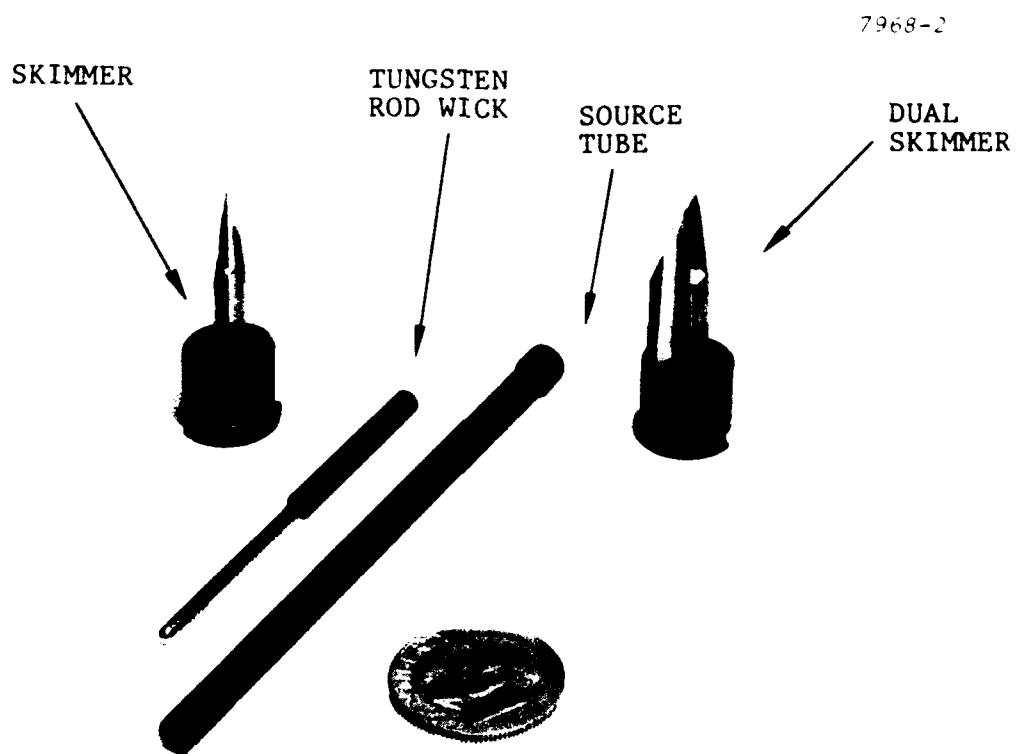


Figure 16. Source Tube, Wick and Skimmers

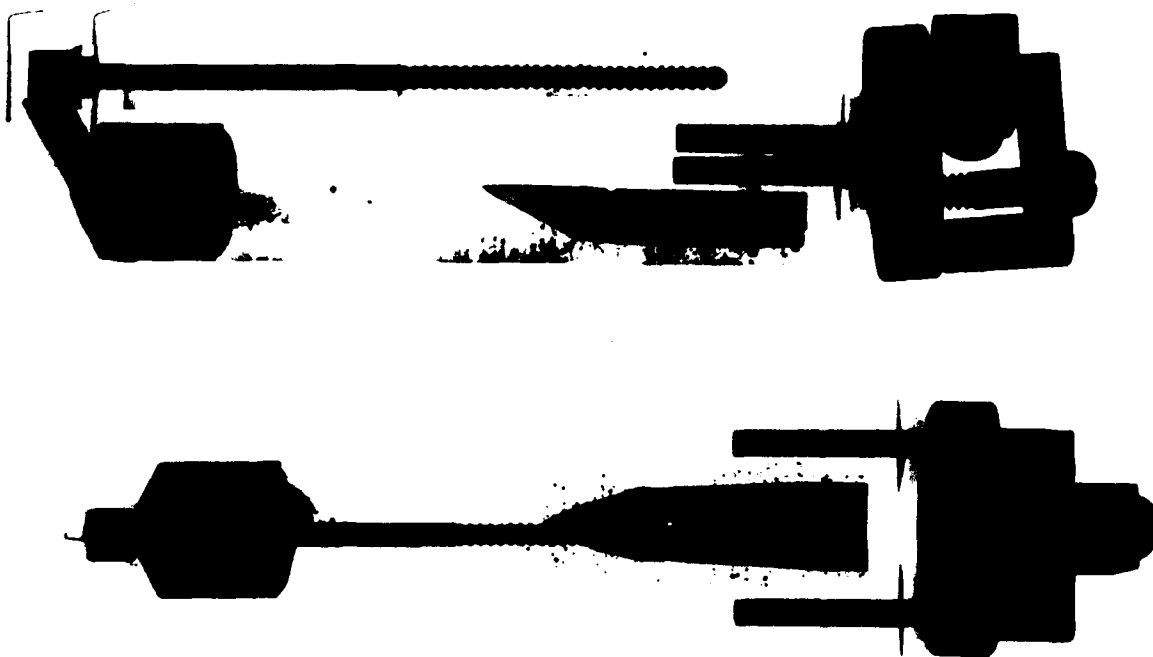


Figure 17. Gold Source, X-ray Photograph

In order to increase the output of the source, empirical parametric studies were conducted. The original source tube aperture was a 0.020-in. diameter "knife" edge type, as suggested by Andres.<sup>4</sup> The aperture diameter was varied from 0.010 to 0.025 in. by successively drilling and countersinking the aperture between runs. No skimmer aperture was used in these experiments, since it was known that the aperture seriously affects the output intensity. The results of variation in aperture diameter are given in Table 1.

During this time, we were plagued with burned out source tubes and plugged skimmer apertures. For these reasons, we could not run long experiments. It was imperative to solve these problems in conjunction with increasing the gold output rate. The following paragraphs will attempt to describe the problems encountered, and the proposed solutions.

Table 1. Effect of Aperture Diameter Variation

Aperture Diameter (in.)	Source Tube Current (A)	Source Tube Temp. (°C)	Deposition* Rate (Å/min)
0.010	80	2260	50
0.015	80	2260	167
0.020	80	2260	269
0.025	80	2260	plugged** skimmer

\*At 7.5-in source tube-to-target distance, and 0.020-in. source tube bore diameter.

\*\*Output rate was probably considerably higher than 269 Å/min in order to plug the aperture.

#### SOURCE TUBE BURNOUT

There were two distinct mechanisms observed that were causing a sublimation of the graphite source tube. One was localized hot spot due to the geometry of the wick and source tube. The other was the gold short circuiting the source tube to the wick, causing a localized hot spot.

#### SOURCE TUBE DESIGN

It was imperative to obtain a relatively long, low temperature gradient heat zone at the center of the source tube in order to vaporize a sufficient amount of gold to maintain equilibrium vapor pressure of about 10-mm Hg, and to minimize hot spots, so that the life of the source tube would be extended.

Initially, the source tube bore was terminated about 1/32 in. above the wick tip, and above the bore the section was solid up to the power clamp. There was a large discontinuity in the heat generation and heat conduction at the interface of the hollow and solid sections. A temperature distribution curve along the source tube axis was made analytically by Andres;<sup>5</sup> two curves are given in Figures 18 and 19. These curves have a relatively short heat zone and a large gradient at the end of the bore. Both empirical and analytical studies indicated that

the wick was conducting a very significant amount of heat out of the source tube. The locus of termination of the wick and that of the top of the crucible, relative to the source tube, are important parameters determining the temperature profile of the source tube.

In order to minimize the electrical and thermal discontinuities of the source tube at the end of the bore, another similar bore was placed into the top section of the source tube. A small plug was left between the top and bottom bores in order to maintain a closed gold vapor chamber. A temperature profile of this hollow source tube is indicated in Figure 20. The heat zone is significantly extended on this curve, and the profile is relatively flat even beyond the termination of the vapor chamber.

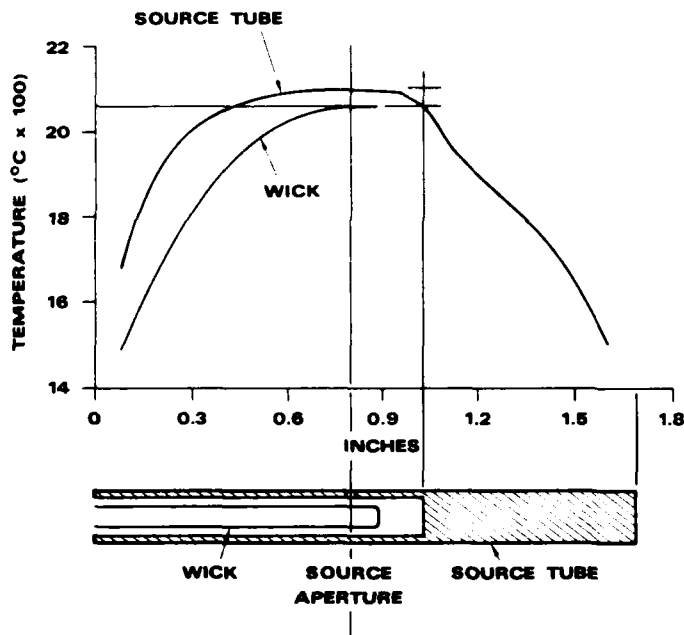


Figure 18. Temperature Distribution Curve, Solid Source Tube, 0.9-in. Wick Insertion Depth

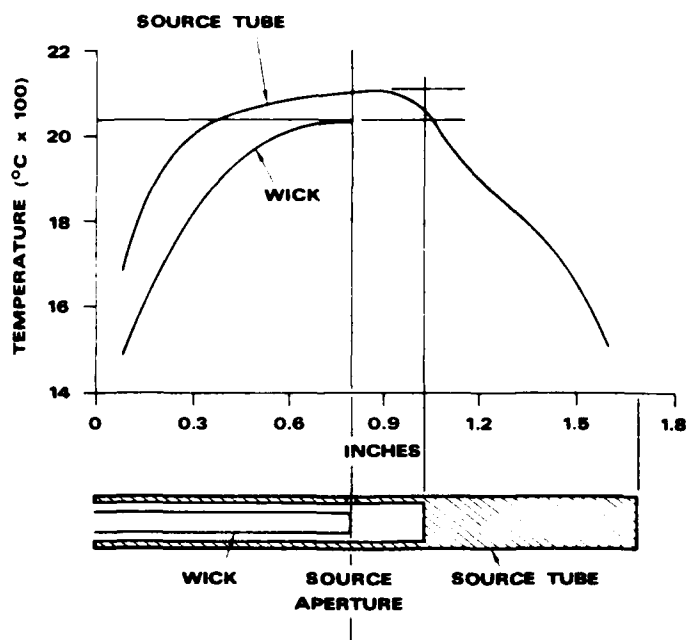


Figure 19. Temperature Distribution Curve, Solid Source Tube, 0.8-in. Wick Insertion Depth

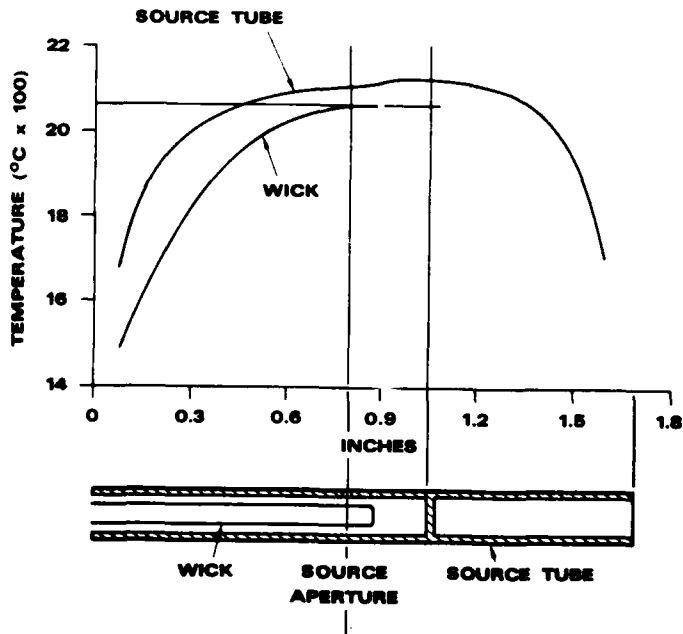


Figure 20. Temperature Distribution Curve, Hollow Source Tube

## WICK DESIGN

In order to further extend the heat zone at the bottom of the source tube, the tungsten wick diameter was reduced to 0.070-in. diameter, and a graphite sleeve was substituted for the removed tungsten. The temperature profile of the slender wick is shown in Figure 21.

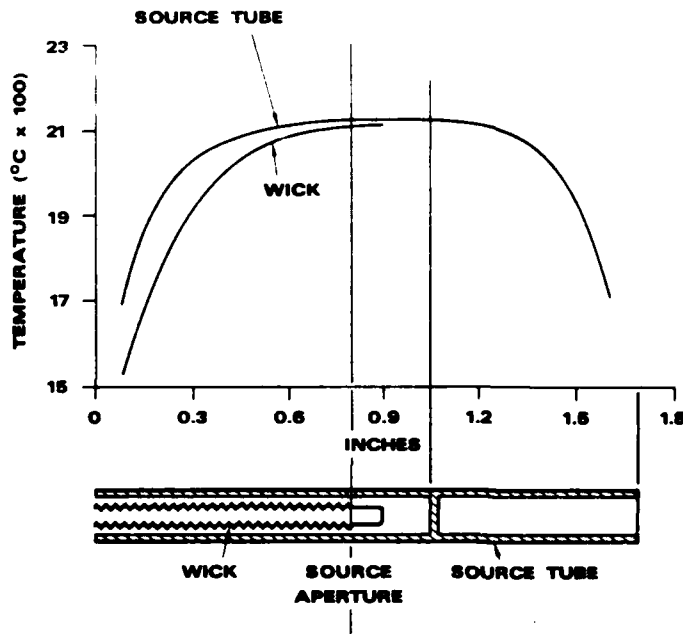


Figure 21. Temperature Distribution Curve, Reduced Tungsten Wick Diameter

The function of the wick is to move the liquid gold from the bottom of the gold reservoir into the heat zone of the source tube. It is important to maintain a high thermal impedance at the wick support in order to maintain all exposed tungsten surfaces at the heat zone temperature, so that gold vapor will not tend to condense at the colder surfaces.

Another problem encountered was the wick-to-source tube electrical short circuit. Gold was condensing at the end of the source tube bore, and this liquid gold was dripping onto the tip of the wick, thereby shorting the wick to the source tube. The short would be generally only momentary, because the source tube current would vaporize the gold droplet. However, repeated current surges across a relatively short section of the source tube would result in the source tube "burnout" due to excessive sublimation of the graphite. An X-ray photograph in Figure 22 indicates gold droplet formation at the end of the bore, in one of the early devices.



Figure 22. X-ray Photograph, Source Showing Gold Droplet at End of Bore

The solution to the shorting problem was obtained by altering three parameters. First, the source tube bore diameter was increased to 0.116 in. from 0.090 in.; the wick diameter was maintained at 0.070 in.



The increased wick-to-bore gap permits the gold droplets to fall to the bottom of the source tube. Second, as indicated previously the solid top shank of the source tube was bored out to 0.116-in. diameter. This resulted in a uniform temperature profile at the upper region of the source tube bore, so that no gold would condense in this region. Third, the gap between the tip of the wick to the end of the source tube bore was increased to 0.060 in. This has the effect of increasing the temperature at the end of the bore, and providing space for larger droplets to form before shorting out to the wick. Small droplets tend to attach themselves to the graphite walls, but as they grow to about 0.030-in. diameter, they drop off. These "small" droplets, of course, will not short the source tube to the wick, for more than a few milliseconds, because they are vaporized by the current surge. It was noted that when cooling the crucible, occasionally gold droplets would still form at the end of the bore; however, they did not constitute a problem.

#### PLUGGED SKIMMER APERTURE

In the early days of the gold source development, there was no problem with the skimmer aperture plugging with excess gold. However, as the source output rate increased, plugged skimmer apertures became a serious problem. The original aperture plates were made of graphite; it was soon discovered that gold tends to "ball up" on the graphite aperture plate. The balls or droplets will grow on the edge of the aperture and eventually form a liquid plug in the aperture. In order to solve this problem, aperture plates of molybdenum were tried, as gold will coat molybdenum evenly. Sufficient length of molybdenum drip leg must be provided below the aperture, so that the liquid gold will drop off before it reaches the aperture. After several days of operation, the molybdenum skimmer aperture plates disintegrated due to gold alloying with the molybdenum. Tungsten skimmer apertures were next tried and are now being used. No damage has been observed on the tungsten skimmer plates after 1200 hours of operation.

#### SKIMMER APERTURE EFFICIENCY

Experimental data indicated that a 1,000-Å/min beam from the source tube, measured at the target 5.5 in. from the source aperture, is degraded to about 300 Å/min by the insertion of a 0.030-in. diameter knife edge planar aperture placed 0.350 in. in front of a 0.020-in. diameter source aperture. The aperture efficiency was, hence, about 30 percent.

In order to improve the aperture efficiency, the 0.003-in. thick aperture plate was bent sharply at the aperture to form a 90° included angle. The aperture efficiency was improved to about 50 percent. The best aperture plates that have evolved were made of 0.003-in. "soft" tungsten sheet, with a 0.030-in. diameter aperture. These plates were suspended over the reservoir, so that the temperature on both sides was considerably above the melting temperature of gold ( $\sim 1400^{\circ}\text{C}$ ). These plates also had long pointed drip legs below the aperture in order to channel the gold flow away from the aperture region.

The output spot has a Gaussian intensity distribution. With a 0.030-in. diameter skimmer aperture, at 5.5-in. target-to-source aperture spacing and a 400-Å/min deposition rate, the spot diameter at the 50 percent drop-off point was about 3/4 in. The required spot size at the 50 percent drop-off point is about 3/8 in., so that there is about 400 percent potential gold consumption rate improvement. An aperture diameter less than 0.025 in., however, at 400-Å/min target deposition rate and standard conditions, will plug up rapidly. This occurs essentially due to liquid gold droplets forming above the skimmer aperture due to the wide angle beam output of the source aperture and flowing into the skimmer aperture. A coarse and a fine skimmer aperture combination will be tried; the coarse aperture should limit the amount of gold reaching the fine aperture plate. Hence, 0.015-in. diameter fine limiting apertures may not plug up; this aperture should give a spot size of about 0.375-in. diameter at the 50 percent drop-off point. It must be kept in mind, however, that the aperture efficiency deteriorates as the aperture size is reduced.

#### RESERVOIR CAPACITY

The reservoir has a capacity of 40 grams of gold. Empirical methods indicate that the gold consumption rate for a 400-Å/min deposition rate on a 0.375-in. diameter (50 percent drop-off) spot is about 0.2 g/h. This will give a 200-h beaming time for the source. Theoretically, this beaming time should plate 6,000 (20 MHz) resonators (30/h). Mechanical shutters are used to stop the gold beam during changing of the resonators in the mask. Between crystal trays, however, the source is put into a standby mode by passing 60 percent (50 A) of the beaming current through the source tube. At this current, the source output rate is less than 2 Å/min, and can be returned to a relatively stable beaming mode in two minutes.

#### BEAM-TO-TARGET ALIGNMENT

Since the gold beam is relatively narrow, in order to obtain optimum efficiency the beam alignment to the target must be within about 0.03 in. The alignment is accomplished by optical means. A 45° prism is mounted on the top of the source tube shield; it is cooperating with a short focus telescope, as shown on Figure 23.

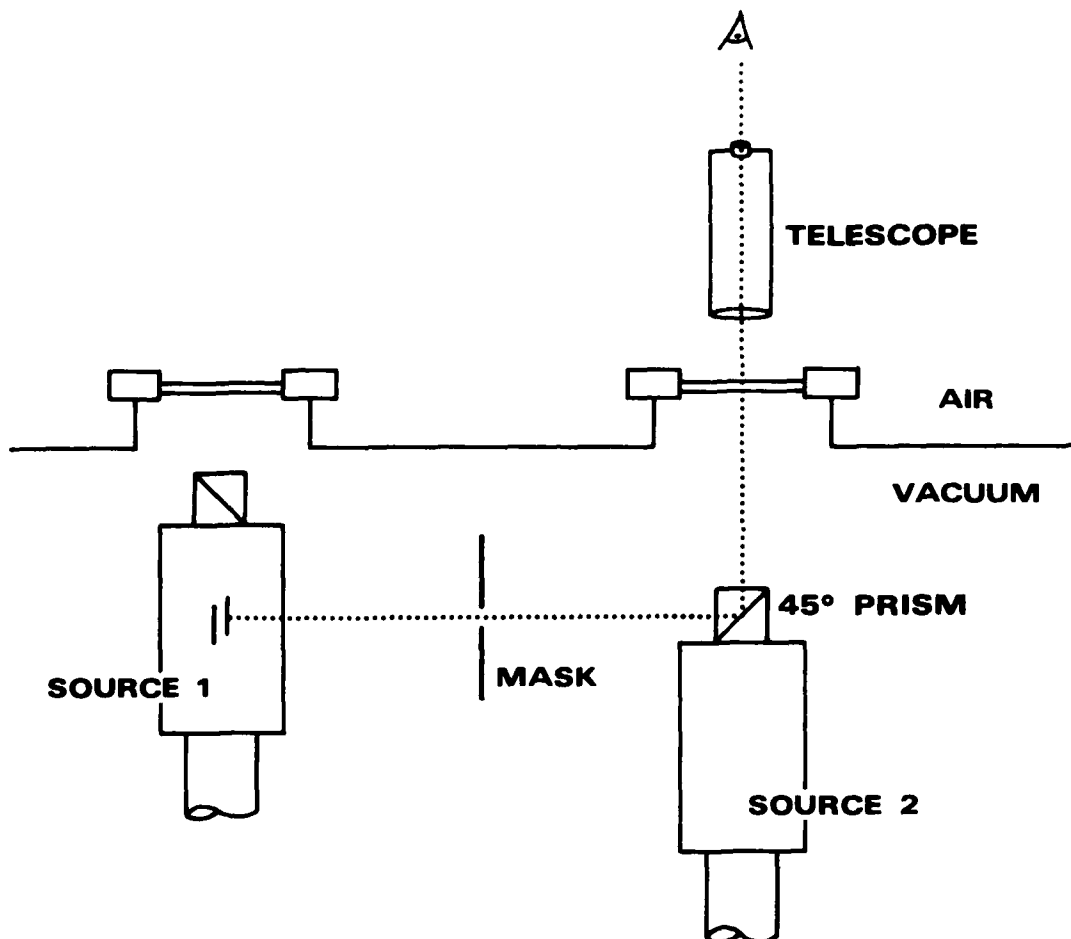


Figure 23. Optical Source Alignment Device

The crystal mask elevator is roughly adjusted so that the center of the mask is in the plane of the center lines of the apertures in the two source tubes. One source tube is energized to the standby mode; the incandescent source tube aperture is now highly visible through the skimmer aperture. The prism, on the top of the other source is adjusted by the elevators to the mask center level. The source aperture is viewed through the mask from the viewport above the prism with a short focus telescope. The source support shaft is rotated and moved in the vertical direction until the source aperture appears centered in the mask and in the skimmer aperture, as viewed through the prism. The elevator is locked, and these positions are noted on the elevator's dial indicators for repositioning. A similar procedure is used to align the opposite source.

Light projected through the skimmer aperture from the incandescent source tube can also be used for rough beam alignment purposes. Fine alignment is obtained from deposition rate monitor crystal data.

In the system, the source-to-mask distance is 5 1/2 in. A relatively large source-to-crystal distance is required for fine plating to minimize the heating effect of the source radiation. The precise dimension was determined by the closest positioning of the six-inch valves on the source and mask elevators. The intensity of the beam varies inversely proportional to the square of the distance between the target and an imaginary beam convergence point behind the source aperture, so that a beam intensity of 2,500 Å/min can be obtained at two-inch source tube-to-target spacing and at the normal operating source tube temperature. At 5.5-in. spacing the intensity is about 400 Å/min.

An important advantage of the nozzle beam source is its relatively low heat radiation characteristics at "fine" tuning. The source is substantially equivalent, in heat radiating characteristics, to a 2,000°C tungsten source of 0.030-in. diameter as seen by the target. The skimmer plate, which contains the 0.030-in. beam limiting aperture, is operating normally at about 1400°C, so that its heat radiation output is insignificant to that of the source tube radiation through the skimmer aperture.

Typically, when the beam shutters are closed, after a one minute "fine" plating process, the final frequency on a 20 MHz fundamental AT-cut resonator will change less than 1 Hz in the plating mask, at the upper turning point.

The nozzle beam sources are enclosed in five concentric cylindrical radiation shields, made of 0.002-in. tungsten foil, and layers of parallel shielding on the top and bottom of the

assembly. The whole device is enclosed in a water-cooled copper jacket in order to minimize the heat radiation onto the mask head.

#### OPERATING LIFE

Two nozzle beam sources were operated at 2,000 to 2,100°C source tube temperature for more than 1,400 hours. Microscopic and X-ray examinations of the crucibles and source tubes indicated no deterioration. It is not known what the failure mechanism of the source is. The present device is expected to operate for several thousand hours. There is no reason to believe that the crucible should ever crack or sublime.

However, the source tube heat zone may vaporize eventually. The source tube diameter and wall thickness may be scaled up if extended life is required. Also, the reservoir size may be scaled up if increased loading capacity is desired. Of course, both of the above modifications will require larger power inputs, and increased cooling capacity.

#### THROUGHPUT RATE

The crystal throughput rate in the system is limited by the coarse deposition rate; this deposition rate, in turn, is limited by the gas evolution from the sources and the heated parts of the chamber and the pumping speed. It appears that the gasses can be driven out of the sources rapidly. However, the pumping speed is not sufficient at  $10^{-7}$  Torr to handle the load for several days after a new source is inserted into the system.

There is only one 6-in. pump provided for both plating stations. It now appears that the addition of a 6-in. pump and the separation of the two stations by a gate valve would be highly desirable. Initial outgassing of the sources, after exposure to atmosphere, is to be accomplished in an auxiliary vacuum system and, after attachment to the system, while still in the elevator well, with the respective gate valve to the plating chamber still in the closed position.

If the chamber and sources have been exposed to the atmosphere, several days of envelope baking with heating tape, and gold source outgassing is required before the pressure reaches  $5 \times 10^{-7}$  Torr at 200-Å/min. deposition rate. Thereafter, the pressure continues to improve with constant daily operation of the system. The pressure at 400 Å/min. deposition rate is typically double that of the 200 Å/min. rate.

The best beaming pressure obtained was  $2 \times 10^{-7}$  Torr, after about two weeks of daily operation. Residual gas analysis data is given on Figure I in the Appendix.

It is imperative to realize that the source aperture to target spacing in this system is 5.5 inches. At a 3.2-inch spacing the deposition rate is increased by about 262 percent, without any increase of plating chamber temperature or any reduction in source life.

It may be possible to redesign the coarse plating chamber, with the use of small format gate valves, so that the source elevator nipple centerline to mask spacing would be about three inches. This will result in deposition times of under one minute for standard 800 Å thick layers, at the present source operating temperatures and pressures. A significant saving in gold usage would also result.

#### ELECTRODE MASK HEAD

The mask head was built according to the guidelines described in the first report (see Figures 24, 25 and 26).

The mask holder contains two contacting pins and the series capacitor. The mask holder may be moved by the manipulator into a special tray. This tray also holds frames with calibrated crystals, and frames with electrical shorts for system calibration purposes.

The contact actuator, mask holder retaining clamp, and the gold beam shutter, are operated by an electro-pneumatic system.

The mask elevator with its mask head, is removable from the plating chamber as an integral unit, and may be bench tested before insertion.

Figure 27 is a block diagram of the plate-to-frequency controls. During actual plating-to-frequency operation, two problem areas have been identified and corrected:

1. Poor sensitivity,
2. Frequency shift due to contact parameter variations at repeated contacting.

The solution to the first problem was to use ceramic insulated coaxial signal leads within the vacuum envelope (not shown on the photographs); initially, only the external signal leads were coaxial.

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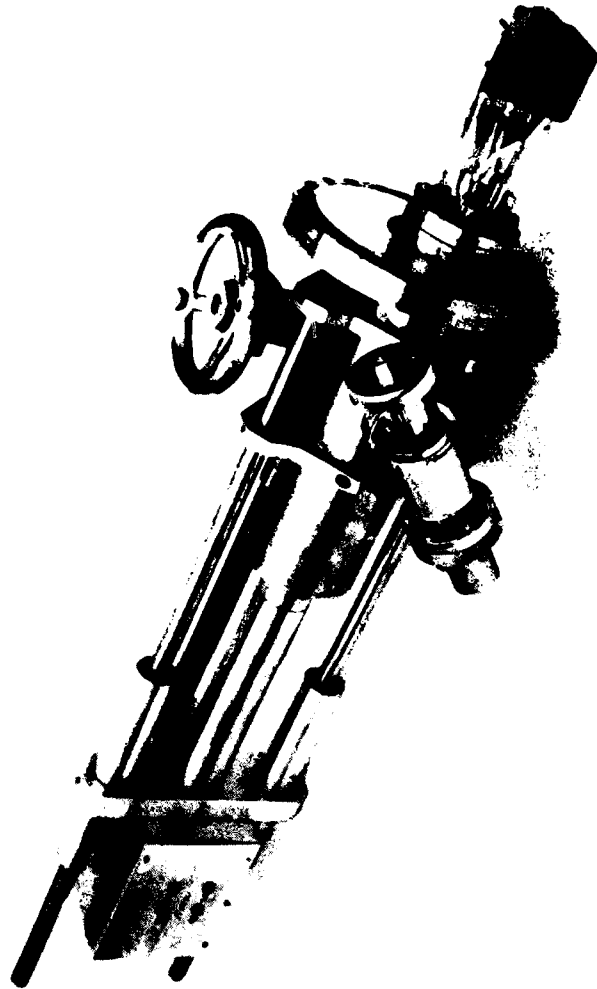


Figure 24. Mask Head and Elevator

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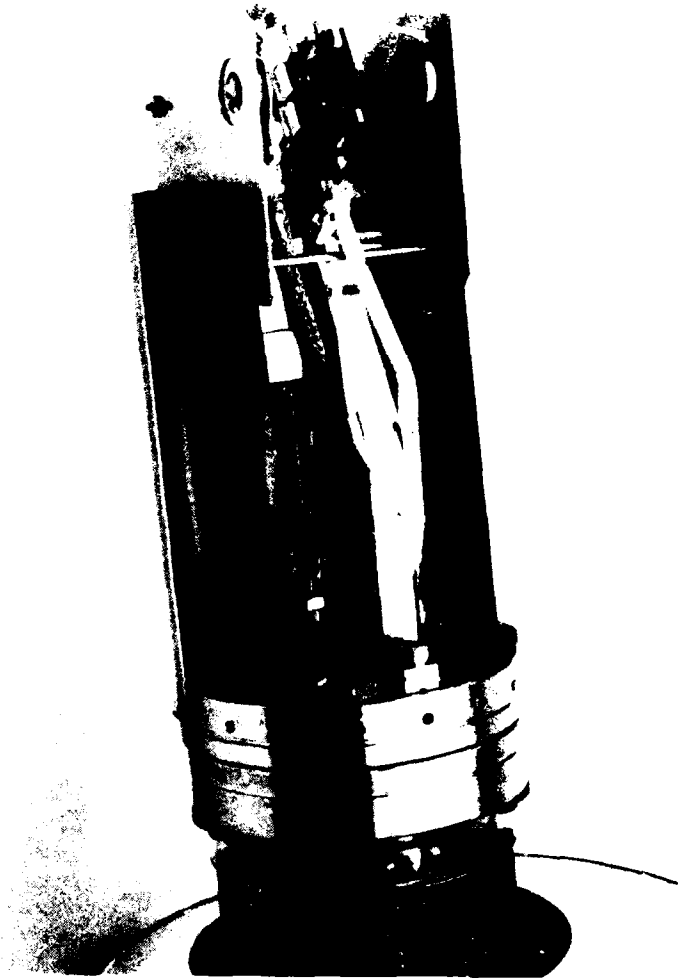


Figure 25. Mask Head



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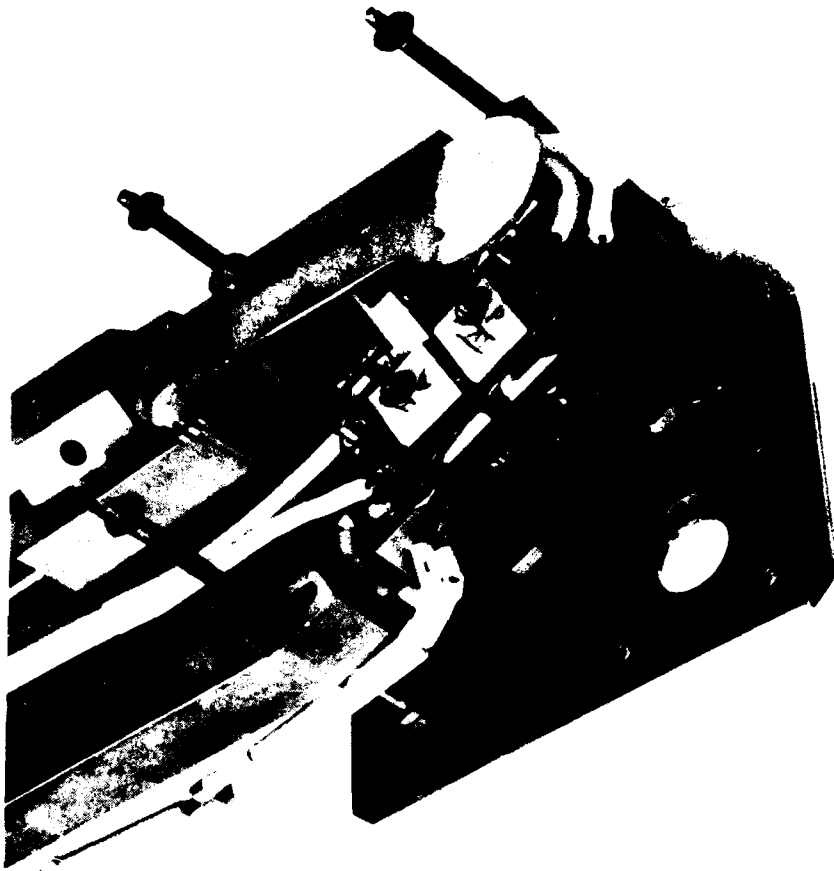


Figure 26. Mask Head Close-up

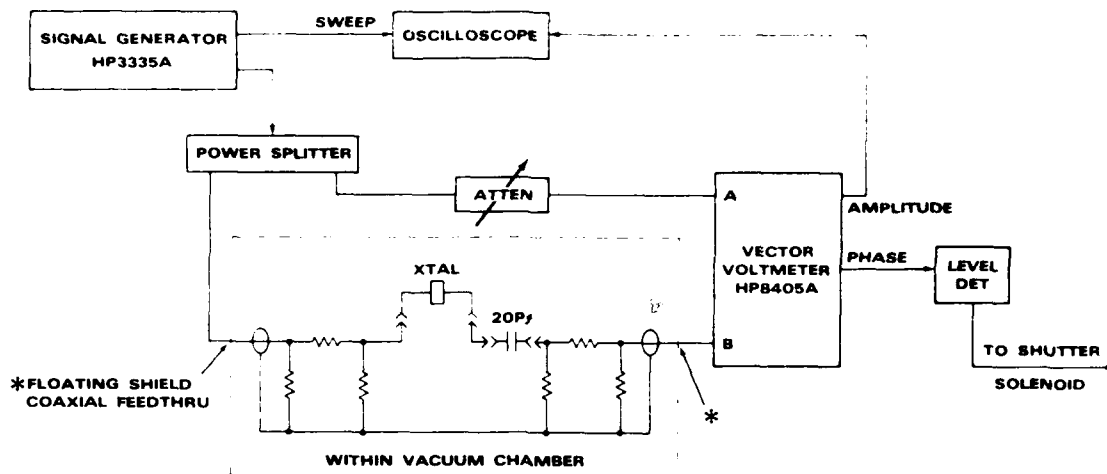


Figure 27. Plate-to-Frequency Controls, Block Diagram

Crystal response to direct feedthrough signal ratios of 5:1 were obtained repeatedly after the above change was made. Ratios of about 80:1 were later obtained by using floating shielded coaxial feedthrus at the vacuum envelope.

The solution to the second problem was found to be an increased contact pressure on the contacting Fernico\* pins.

It was not conclusively proven why the contact pressure would change the frequency indication by as much as 200 Hz. It was theorized that a surface film at the Fernico-to-gold interface had to be "broken through," in order to maintain the circuit resistance within a tolerable range.

Frequency indication variations of no larger than 10 Hz are obtained with the increased contacting force.

#### SEALING FIXTURE

The heater chamber and hydraulic ram schematic is shown in Figure 28. The sealing fixture was built as described in the first report (see Figures 29 through 31). Metal bellows were used to obtain the vacuum seal between the stationary and the moving components.

\*Trademark, General Electric Company

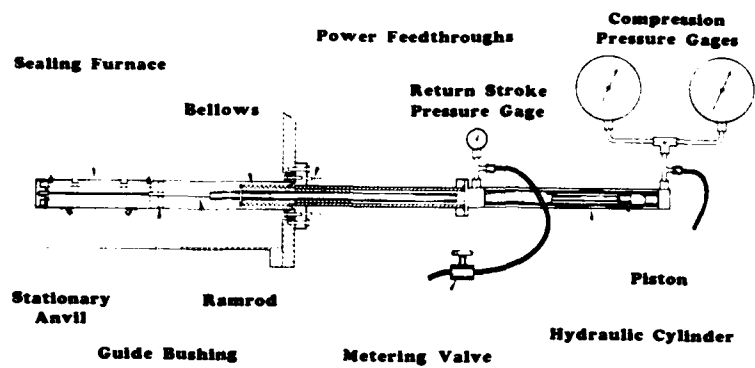


Figure 28. Sealing Fixture Schematic

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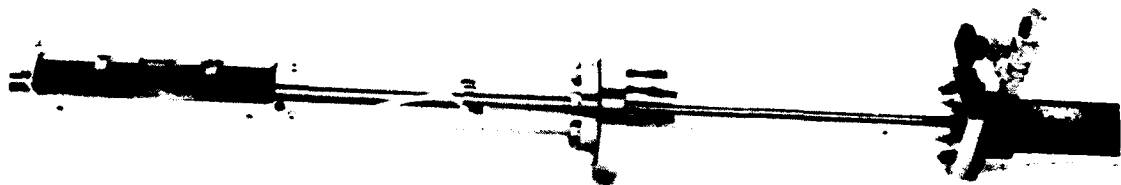


Figure 29. Sealing Fixture

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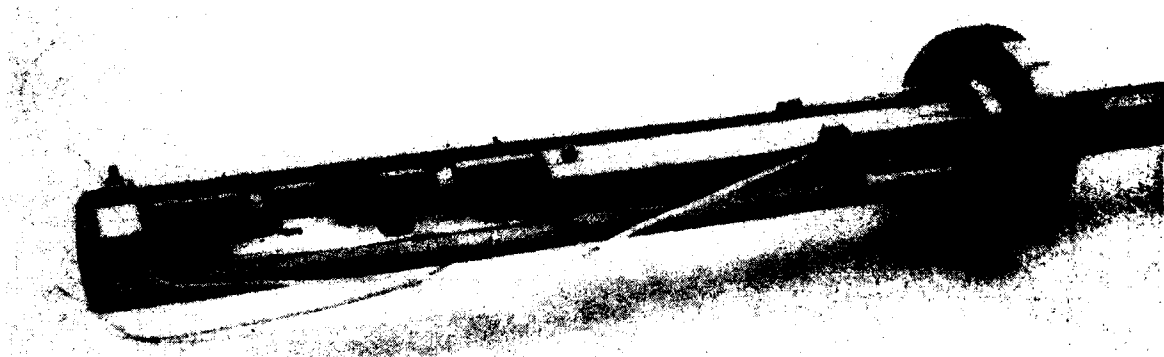


Figure 30. Sealing Fixture, Showing Sealing Furnace and Ramrod

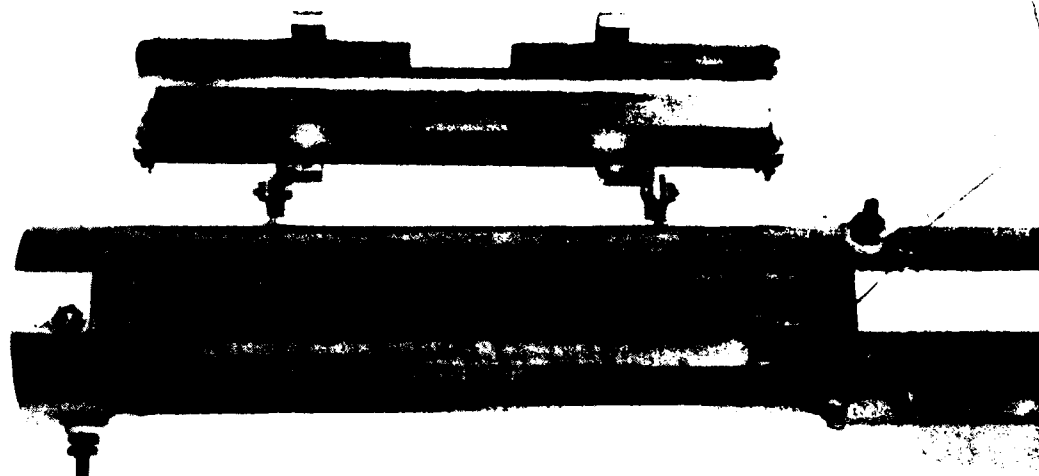


Figure 31. Sealing Fixture, Sealing Furnace

The ram is capable of exerting a total force of 2,000 lb on the stationary anvil. The ramrod that enters the crystal tray is made of molybdenum in order to obtain maximum resistance to buckling, since its maximum diameter is limited to the crystal frame width (0.400 in.).

An internal copper bushing was retrofitted into the sealing oven in order to guide and laterally support the ramrod at the crystal tray during the compression cycle.

The initially used Nichrome\* heaters were coating the heater core ceramics with a layer of metal, and shorting out coils. The Nichrome wire was replaced with tantalum wire and no further problems were observed.

\*Trademark, Driver-Harris Co.

The sealing oven has three heating elements enclosed in copper bars. Two of the bars are mounted on the main oven envelope; the third is mounted on the door of the oven. Even though the heater bars are well-isolated thermally from the oven envelope, the heater on the door runs much hotter than the other two heaters. Installation of an independent current controlling device was required.

The multiple or stack thermocompression sealing process works well. Data taken on 20 MHz packages with ten units sealed simultaneously resulted in a yield of 83 percent at Radiflo leak testing for all causes, including defective parts. This data was taken on a sample size of 450.

One sealing run was made recently, with twenty envelopes sealed simultaneously. All units passed Radiflo leak detection.

#### ELEVATORS

Elevators were utilized on the source and mask heads, so that they are retractable behind a gate valve for maintenance purposes without losing plating chamber vacuum. The elevator shaft-to-housing space is sealed with metal bellows and the elevator is driven by a manually operated lead screw. The elevator chamber is rough-pumped prior to the insertion of the head into the plating chamber.

#### CONTROL CIRCUITRY

The ERADCOM Quartz Crystal Fabrication Facility vacuum chamber is divided into five sections: entrance, clean and bake, plating, seal, and exit. The vacuum system controls are housed in cabinets separated and individualized for each section of the system. The cabinets are located directly across the vacuum chamber in front of each operator station (photo, Figure 2). Operators have a clear view of all indicators, lights, etc. However, operators are not able to reach these cabinets across the vacuum system due to the clearance left for servicing the vacuum system. Therefore, all switches and controls that an operator would normally adjust on a regular basis are provided as remote controls located on a small panel within easy reach at each of five operator stations.

Each of the cabinets housing the facility controls contains a compressor for a cryogenic vacuum pump and a vacuum pressure gage. The remaining controls are individualized for each section providing, where needed, such functions as: valve control, power to uv and incandescent lamps, power to heaters, power to the gold source evaporators, and controls for mask head operation. Closed loop temperature regulators with thermocouple sensors are provided for heater control. Many functions such as heater power, valve operation, etc. are interlocked with cooling water flow, vacuum pressure or other suitable protective criteria to minimize problems due to operator error. While the facility controls presently are intended for manual operation, the possibility of future computer automation has been considered and circuit layout and other features were designed to minimize the difficulty of adding this capability.

A sixth cabinet (shown at the extreme left of Figure 2) houses equipment used in automatically plating crystals to a specified frequency. This cabinet is on rollers and can be moved along the length of the vacuum chamber to either the rough or fine crystal plating positions. A block diagram of the plate-to-frequency controls is shown in Figure 27. A resistive Pi-network is mounted within the vacuum chamber as an integral part of the rough and the fine plating mask head assemblies. This is nominally a 25- $\Omega$  network constructed by metallization and film disposition on ceramic substrates. The load capacitor may be added or omitted.

The network is balanced to zero phase with a short substituted for the crystal. Crystals are plated until the phase is again zero, with the frequency from the generator adjusted to the desired crystal operating frequency.

The Hewlett-Packard HP3335A signal generator provides a great number of features that make setting up the system and/or checking the crystals for proper operation quite easy. For example, seven preset combinations of control settings can be stored in the instrument memory and instantly recalled at any time; a wide range of automatic or manual sweep conditions is available.

Automatic plating-to-frequency is accomplished simply by the following steps: first, setting the signal generator to the desired crystal frequency; second, pressing the shutter open button; and third, waiting for automatic closure of the shutter that blocks gold deposition.

Emergency power off buttons are located at the front and back of all control cabinets to provide for equipment and/or personnel protection.

## CONCLUSIONS

1. The basic design of a high throughput high vacuum processing system for precision quartz resonator units has been proven with an operational device. Each work station was operated at the rate of 15 units per hour, although the entire system has not been operated at that rate so far.

With the use of 25 position trays, a nominal production rate of 25 units per hour should be realized.

However, because of the complexity of this facility, an integrated test of the entire processing system will be required to establish operating reliability of each work station and total system capacity.

2. A highly reliable, long-life, nozzle beam gold source was developed. The source is capable of operating for two hundred hours at deposition rates of 2,500 Å/min at 2.0 inches of "throw" distance, with one charge of gold. For 20 MHz fundamental resonators, this typically corresponds to 2,000 + units plated, (on one side). The deposition rate is characterized by extremely high short-term stability, after the initial warmup period.

This basic design may be scaled up or down for other specific applications.

3. The system was operated for several months at a throughput of about one tray of 10 crystal units daily without a failure, except for the failure of the crystal unit contacting pins. The contact pins have been redesigned, and no further problems are expected.
4. The major source of gas in the plating chamber is the gold sources, not the chamber walls, even when the walls are heated. Chamber pressure of  $5 \times 10^{-7}$  Torr was obtained after 8 hours of source operation at a 400-Å/min deposition rate at 5.5-inch throw distance. The pressure typically reaches equilibrium at under  $5 \times 10^{-7}$  Torr in one to two weeks of operation.
5. There was insufficient time available in this project to optimize the efficiency of the gold utilization, i.e., to reduce the beam spot size to the crystal plate size. A 400 percent improvement in the gold utilization may be achievable. This would reduce the loading downtime by a factor of 4, and would reduce the gold charge requirement by a similar factor.

## RECOMMENDATIONS

1. Install a valved off separately pumped cooling chamber between the two plating stations. This would permit the cooling of the crystal units at a pressure about two decades lower than that of the coarse plating chamber. The pressure in the fine plating chamber could be maintained at about one decade below the coarse plating chamber. An additional pump on the fine plating chamber would be required.
2. The QXFF is potentially capable of production rates of about 50 units per hour. The major limiting factor now is the coarse plating speed. In order to achieve the above rate, a new coarse plating chamber is required, with the source head to mask head centerline spacing of three inches. This can be achieved with the use of "close format" gate valves on the elevator tubes. The addition of the above cooling chamber, and the extension of some ovens is necessary to achieve the increased production rate of the system. It should be noted that maintenance downtime is not considered in the "potential" production rate.
3. It may be possible to achieve significant improvements in the nozzle beam gold source, with a relatively modest program. Larger beaming rates could certainly be achieved with larger source tubes and wicks. Reducing the size of the beam may be achieved by multiple skimmer apertures. Improved water cooled shields should be tried, particularly with the higher power units.



#### ACKNOWLEDGMENTS

The author wishes to express his appreciation for the tireless efforts of Dr. E. Hafner of the U. S. Army ERADCOM for providing guidance and counsel from his initial concept through the construction of the Quartz Crystal Fabrication Facility.

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Sincere appreciation is expressed for the outstanding design work, initiative, and inexhaustible patience of Wade Roberts, of the Engineering Design Definition Group.

The author also wishes to express his gratitude to Sanborn Hutchins, the toolmaker's toolmaker at GEND Maintenance Tool Room, who made real parts out of nearly impossible design geometry and materials.

Last, but not least, the author is indebted to J. Havelka, GEND Crystal Resonator Development group, for running the nozzle beam source experiments, for assembling and operating the facility, and for making valuable suggestions on both devices.

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APPENDIX  
PLOT OF EQUATION TWO FOR GOLD

Figure I. SLA QXFF RGA Data

Gas Species	Mass	Mass Peak Heights (Amperes)			
		Rough Plating		Sealing	
		Room Temp.	Processing	Room Temp.	Processing
H <sub>2</sub>	2	1.0 x 10 <sup>-7</sup>	3.9 x 10 <sup>-7</sup>	5.6 x 10 <sup>-9</sup>	1.9 x 10 <sup>-8</sup>
CH <sub>4</sub>	16	1.2 x 10 <sup>-9</sup>	7.3 x 10 <sup>-9</sup>	1.2 x 10 <sup>-10</sup>	9.0 x 10 <sup>-10</sup>
H <sub>2</sub> O	18	2.8 x 10 <sup>-9</sup>	4.9 x 10 <sup>-9</sup>	2.6 x 10 <sup>-10</sup>	4.4 x 10 <sup>-9</sup>
CO/N <sub>2</sub>	28	5.7 x 10 <sup>-9</sup>	8.5 x 10 <sup>-8</sup>	1.4 x 10 <sup>-9</sup>	4.5 x 10 <sup>-9</sup>
O <sub>2</sub>	32	2 x 10 <sup>-11</sup>	2 x 10 <sup>-11</sup>	1.2 x 10 <sup>-11</sup>	2.0 x 10 <sup>-11</sup>
Ar	40	6.5 x 10 <sup>-10</sup>	4.2 x 10 <sup>-9</sup>	6.4 x 10 <sup>-11</sup>	2.1 x 10 <sup>-10</sup>
CO <sub>2</sub>	44	2.3 x 10 <sup>-10</sup>	1.4 x 10 <sup>-9</sup>	1.2 x 10 <sup>-11</sup>	2.2 x 10 <sup>-10</sup>
Total Chamber Pressure (Torr) (Ion Gage)		4.2 x 10 <sup>-9</sup>	3.6 x 10 <sup>-7</sup>	6.3 x 10 <sup>-8</sup>	2.0 x 10 <sup>-7</sup>

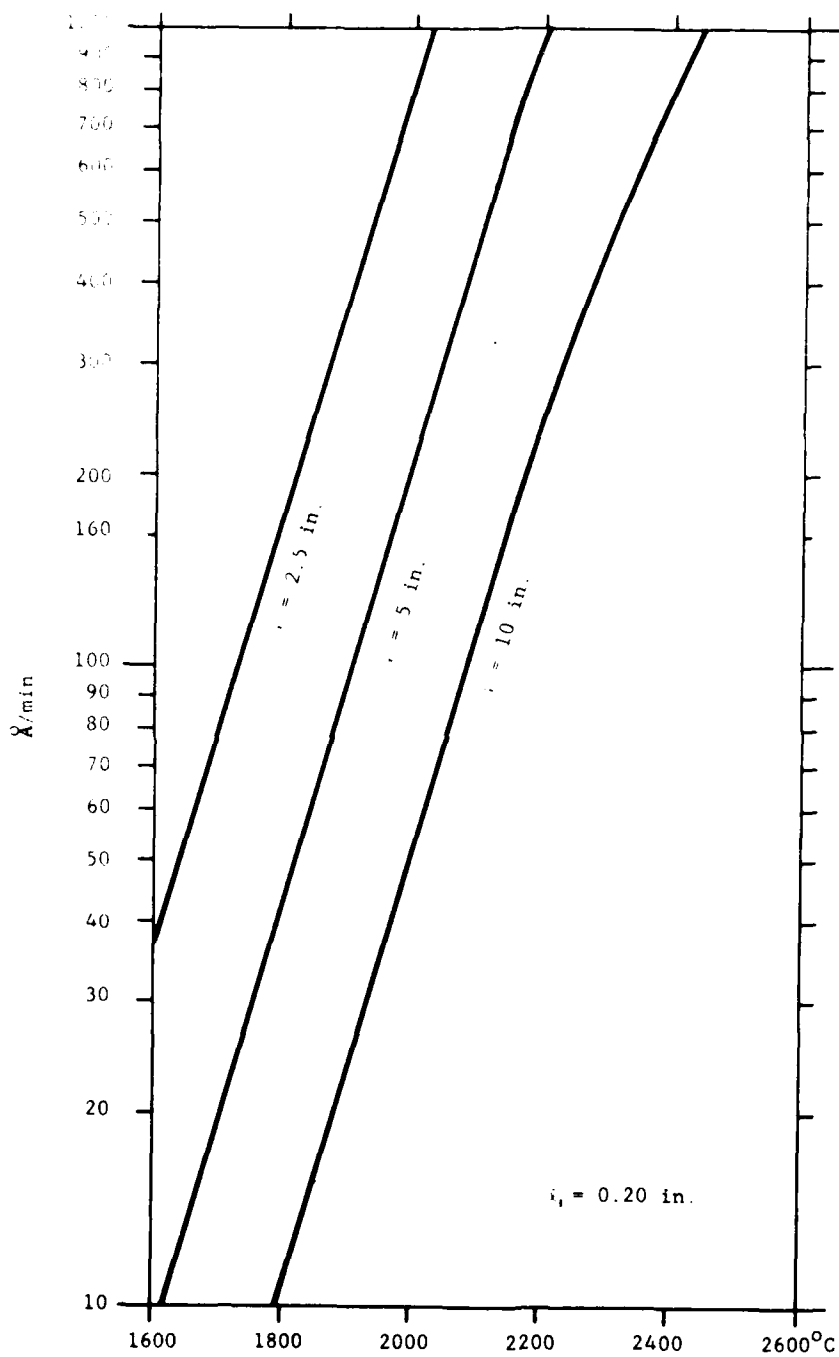


Figure II. Ideal Source Behavior - Rate of Gold Deposition

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